IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of Masaki YANAGIOKA
Application No.: 10/599,151
Filed: September 21, 2006
For: RUBBER COMPOSITION FOR TIRE TREAD AND PNEUMATIC TIRE USING THE SAME
Group Art Unit: 1796
Examiner: John Uselding
Confirmation No.: 5036

DECLARATION UNDER 37 C.F.R. § 1.132

I, Masaki Yanagioka, declare that:

I am the inventor of the above-captioned patent application.

I received my Master of Engineering from the University of Tokyo in 2001, and I have been employed by Bridgestone Corporation since 2001, where I have been engaged mainly in research and development of fillers for a tire. Further, I received my Ph.D. of Chemical Engineering from Stanford University in 2009.

I have made the following experiments in order to measure a hydrogen desorption ratio and a toluene tinting permeability of the carbon black in Examples 4-7 of Sakakibara (US 6,197,870) and further evaluate wear resistance of a tire comprising a tread portion made from a rubber composition compounded with the carbon black in Example 5 of Sakakibara (US 6,197,870).

Experimental Procedure

In the experiments, I used a carbon black producing furnace comprising:

a combustible fluid introducing chamber (inner diameter: 450 mmϕ, length: 400 mm),

an oxygen-containing gas introducing cylinder (inner diameter: 250 mmϕ, length: 300 mm) having a straightening vane for distributing an oxygen-containing gas introduced from an outer periphery of a furnace top portion into the combustible fluid introducing chamber,

a fuel oil introducing apparatus disposed on a center axis of the oxygen-containing gas introducing cylinder,

a convergence chamber (inner diameter at upstream side: 370 mmϕ, inner diameter at downstream side: 80 mmϕ, converging angle: 5.3°) gradually converging the cylinder,

a stock oil introducing chamber including four stock oil spraying ports separately formed at the downstream side of the convergence chamber, and

a reaction continuing and cooling chamber (inner diameter: 160 mmϕ, length: 7500 mm) having a reaction chamber and 25 spray devices for pumping a quenching water for the stop of the reaction at the downstream side of the stock oil introducing chamber.

Further, the carbon black producing furnace has a structure capable of inserting thermocouples into the furnace at arbitrary several places for monitoring the temperature inside the furnace.

In the experiments, a fuel oil A having a specific gravity of 0.8622 (15°C/4°C) is used as a fuel, and a heavy oil having properties shown in Table 1 is used as a starting oil.

Table 1

Specific gravity (JIS K2249) (15/4°C)		1.1319
Kinematic viscosity (JIS K2283) (mm^2/s at 50°C)		26.7
Water content (JIS K2275) (%)		0.5
Residual carbon (JIS K2210) (%)		11.6
Sulfur content (JIS K2213) (%)		0.4
Carbon content (%)		90.1
Hydrogen content (%)		5.4
BMCI *1		160
Distillation characteristics (°C)	I.B.P. *2	188
	10% cut point	234
	30% cut point	291
	50% cut point	360

*1 BMCI: Bureau of Mines Correlation Index

*2 I.B.P.: Initial Boiling Point

The carbon blacks in Examples 4-7 of US 6,197,870 are presently prepared in the above-described carbon black producing furnace under the operating conditions shown in the following Table A. With respect to the resulting carbon blacks, the hydrogen desorption ratio and the toluene tinting permeability are measured according to the methods described in the specification of the present application. Results are also shown in the following table A.

Table A: Operating conditions and Results

	Carbon black prepared according to Example 4 of US 6,197,870	Carbon black prepared according to Example 5 of US 6,197,870	Carbon black prepared according to Example 6 of US 6,197,870	Carbon black prepared according to Example 7 of US 6,197,870
Total amount of air supply (Nm^3/hr)	2300	1700	1500	1800
Amount of fuel oil supply (Nm^3/hr)	134	94	74	105
Fuel atomized air (Nm^3/hr)	150	150	150	150
Fuel combustion rate (%)	170	180	200	170
Amount of raw material oil at first stage (kg/hr)	295	207	178	183
Amount of raw material oil at second stage (kg/hr)	228	147	115	122
Reaction time (mmsec.)	96	280	357	340
N_2SA (m^2/g)	131	162	194	252
IA (mg/g)	118	173	207	270
CTAB surface area (m^2/g)	121	146	170	200
N_2SA / IA	1.11	0.94	0.94	0.93
$0.260 - 6.25\times10^{-4}\times$(CTAB) (%)	0.18	0.17	0.15	0.14
Hydrogen desorption ratio (%)	0.32	0.16	0.12	0.10
Toluene tinting permeability (%)	60	98	100	100

<Preparation of rubber composition>

A rubber composition is prepared according to a compounding recipe shown in Table 4 by using the carbon black prepared according to Example 5 of US 6,197,870 in a Banbury mixer. This rubber composition is vulcanized in a pressure type vulcanizing apparatus at a temperature of 145°C for 30 minutes.

Table 4

Compounding recipe (parts by weight)	NR *3	50
	BR *4	50
	Carbon black	50
	Antioxidant 6PPD *5	1
	Stearic acid	2
	Zinc oxide	3
	Vulcanization accelerator BBS *6	0.8
	Vulcanization accelerator DPG *7	0.2
	Sulfur	1
Tire size		11R22.5

*3 RSS#3

*4 cis type BR01, made by JSR Corporation

*5 Nocrac 6C, made by Ohuchi-Shinko Chemical Industrial Co., Ltd. N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine

*6 made by Ohuchi-Shinko Chemical Industrial Co., Ltd. N-t-butyl-2-benzothiazol sulfenamide

*7 made by Sumitomo Chemical Industrial Co., Ltd. diphenylguanidine

<Evaluation of Wear resistance>

A tire for a truck comprising a tread portion made from the rubber composition compounded with the carbon black prepared according to Example 5 of US 6,197,870 is prepared and mounted onto a vehicle. At a time of running over 20,000 km, a decreasing amount of a groove is measured and represented by an index on the basis that Comparative example 1 disclosed in the present specification is 100. Concretely, a loss amount through wearing is measured by means of a Lambourn abrasion tester, from which an index of the wear resistance is calculated according to the following equation.

Wear resistance = loss amount of a rubber test piece as a comparison standard through wearing/loss amount of a rubber piece to be tested through wearing × 100

The larger the index value, the better the wear resistance.

As a result, the tire comprising the tread portion made from the rubber composition compounded with the carbon black prepared according to Example 5 of US 6,197,870 has a wear resistance index value of 90.

Summary

As seen from Table A, the carbon blacks prepared according to Examples 4-7 of US 6,197,870 do not satisfy either of (1) a hydrogen desorption ratio > 0.260 - $6.25\times10^{-4}\times$CATB (wt%) or (2) a toluene tinting permeability of not less than 90%. Therefore, Sakakibara (US 6,197,870) fails to disclose the carbon black used in the present invention.

As further seen from the above result, the carbon black prepared according to Example 5 of US 6,197,870 has a toluene tinting permeability of 98% and a hydrogen desorption ratio of 0.16% and this hydrogen desorption ratio is very close to 0.260 - $6.25\times10^{-4}\times$CATB (wt%), but the wear resistance of the tire comprising the tread portion made from the rubber composition compounded with the carbon black presently prepared according to Example 5 of US 6,197,870 is notably deteriorated as compared to Comparative example 1 disclosed in the present specification

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under § 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: February 22, 2010 Declarant: Masaki

Masaki Yanagioka